BOSTON MEDICAL LIBRARY AUG 2 1913

THE ANALYST.

JANUARY, 1895.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS.

The usual Monthly Meeting of this Society was held on December 5th at the rooms of the Chemical Society, Burlington House. In the absence of the President, Mr. Otto Hehner took the chair.

The minutes of the last meeting were read and confirmed.

H. S. Shorthouse, Pershore Road, Birmingham, was proposed for election as member.

The following gentlemen were duly elected: as member, Raymond Ross, Fort Royal, Worcester; as associates, C. A. Mitchell, M.A., and J. Lewin.

On the proposal of Mr. Allen, seconded by Mr. Smetham, Mr. B. Kitto and Dr. F. L. Teed were appointed auditors.

Mr. Richmond then read the following paper:

NOTE ON RECKNAGEL'S PHENOMENON.

By H. Droop Richmond.

Recknagel first observed that the specific gravity of milk, when taken as soon as possible after milking, was lower than observations on the same milk at later periods. He attributed this to a swelling of the casein, though it should be more properly a contraction of the casein.

Vieth (Analyst, xiv., 69) made a series of experiments, and found a rise in twenty-four hours varying from 0·001 to 0·002, and averaging 0·0013, and confirmed Recknagel's statement completely.

Bourcart (*Bull. Soc. Chim.* [3], i., 24) has also made the same observation.

Pappel and I (*Journ. Chem. Soc.*, lvii., 754) have observed in the milk of the gamoose an average rise of 0·0007 in twenty-four hours, and concluded that the rise was not so great in the milk of the gamoose as in that of the cow. The maximum rise observed in twenty-four hours was 0·0009, and the minimum 0·0003. In one sample examined later than the others, I found a decrease in specific gravity, the figures being 1·0351 after two hours, and 1·0345 after twenty-four hours. The duplicate determinations with different pynometers only differed by 0·00002, and I do not think that experimental error was the cause of the difference.

The American Association of Official Agricultural Analysts proposed to touch upon this subject for this year, but their report has not yet reached me. In private communications both Dr. Babcock and Dr. Farrington have informed me that they have either observed no rise or a very slight one.

It appeared to me desirable to further examine the question, and to see if the phenomenon were general, or, if not, under what circumstances it took place, and to determine to what change in the constituents it was due.

The following reasons are possible:

(i.) When fresh milk is drawn from the udder, minute air-bubbles form, and are suspended in the milk for a long period.

(ii.) Milk-sugar in the udder may be formed as anhydrous sugar, and the rise may be due to a hydration.

(iii.) A change in the albuminoid, analogous to that of fibrin, may occur; in this case it would probably be enzymic.

(iv.) The fat globules may contract very slowly as the milk cools, owing to their being surrounded by a layer of badly conducting material.

(v.) A chemical change, such as that between aldehyde and water (cf. Perkin, *Journ. Chem. Soc.*, li., p. 817), may take place.

The reasons may be examined as follows:

(i.) This reason is not very likely, as microscopic examination with a high power fails to show air-bubbles after about one hour after milking; froth produced by artificial means disappears in about one hour or less.

(ii.) This is also most improbable, as milk-sugar solutions do not change in density during chemical change indicated by a rapid variation in specific rotation; nor does the milk-sugar, estimated by the polariscope, differ when estimated soon after milking, and after the lapse of some hours, though a rise in the specific gravity is taking place.

(iii.) Salicylic acid has an inhibitive action on many enzymes; preliminary experiments have given indications of the rise being hindered by salicylic acid.

(iv.) Is also improbable, as in this case milks rich in fat should show more rise than poor milks; experiment does not bear this out.

(v.) Chemical change should follow Harcourt and Esson's laws. Data are not sufficiently numerous to ascertain whether the rise in specific gravity does so, but there are indications that, though in fair agreement, the rise is not wholly in accordance with them.

O'Sullivan and Tompson (*Journ. Chem. Soc.*, lvii., 865) have shown that an enzyme, invertase, also follows to a great extent Harcourt and Esson's laws, and that therefore there is no sharp distinction between chemical and enzymic action.

It seems probable, therefore, that Recknagel's explanation that a change in the casein occurs is correct, and that this is due to an enzyme (possibly the enzyme which causes the change within the udder); it would appear from this that milk is still a living tissue after secretion. It is probable, then, that fore-milk (*i.e*, milk that has remained ready formed for some time within the udder) will show much less rise than the later portions (which are drawn away as secreted). Preliminary experiments have shown indications of this being the case.

I have shown that for two successive years practically the same differences have occurred between the fat calculated and that estimated at different periods of the year (ANALYST, xix., 82); though I hesitated to place too much reliance

on the apparent concordance, it is interesting to bear this in mind when studying the periodical variations of the Recknagel phenomenon. There is some evidence already that the rise in specific gravity takes place to a much greater extent in the autumn and winter than in the spring and summer, and that the year can be divided into periods corresponding to those indicated by the agreement of the calculated fat. Much systematic work will, however, be necessary to establish the views that I have indicated.

I reserve quantitative details for a later communication, but I may say that while, in some cases, the rise has been as great as that found by Recknagel and Vieth, in others it has been absolutely nil.

Discussion.

Mr. Alfred Smetham said that in a series of milk analyses which he had conducted in connection with some experiments in Cheshire cheese-making, there had occurred considerable differences between the percentages of fat as calculated from the specific gravities (which were taken at the farm where the cheese was being made) and the amounts arrived at from determinations made in his laboratory by the Adam's process. These differences were so marked as to cause him to distrust the hydrometer with which, in the first instance, the specific gravities were taken; but the instrument, when tested, was found to be quite correct; and, moreover, the discrepancies still remained when a balance had been substituted for the hydrometer. A rise in the specific gravities of the samples seemed to have taken place during the time which elapsed between the measurements at the farm (which circumstances rendered it necessary to make within half an hour of milking) and the analyses made in the laboratory. After careful observation and consideration, he came to the conclusion that this must be due to the gas, as of the milk and the air bubbles mechanically carried into the milk during the operation of milking, which would disperse after some lapse of time. The experiments extended over several months of two successive years, but were not continuous, taking place in the late summer and autumn only of each year.

Dr. Sykes said he thought that enzymic action might possibly account for the phenomenon under consideration. It was now a well-ascertained fact that the peptonization of proteids was a process of hydrolysis, and this would no doubt be accompanied by a rise in the specific gravity of the solution of the proteid, just as a solution of cane-sugar, owing to fixation of water by the sugar molecule, becomes specifically heavier under the action of the enzyme invertase.

In the absence of the respective authors, Dr. Dyer read a paper entitled "Note on Woody Fibre Determination," by A. P. Aitken, D.Sc.; also "Note on a Sample of Red Water," by W. F. Lowe; and the three following papers :

THE ANALYSIS OF SODIUM PEROXIDE.

By L. Archbutt, F.I.C.

Shortly after sodium peroxide became an article of commerce, I had occasion to make a full analysis of a sample of it, which led to some observations of interest. As I am not aware that a really accurate, rapid method for the assay of this article has been published, I venture to bring the results of my experiments before the Society.

The sample of the peroxide which I examined was taken from a full 14-lb. canister fresh from the makers' works. A portion of it was transferred to a stoppered tube, and all the weighings were made by taking some from the tube, which was immediately re-stoppered and the loss of weight ascertained.

Estimation of Soda (Na_2O).

About 0·8 gramme was accurately weighed, dissolved in water, boiled to decompose the peroxide, cooled, tinted with methyl orange, and titrated with normal sulphuric acid. A few drops more of the acid were then added, the solution evaporated to dryness in a tared platinum dish, and the residue weighed after ignition. From the weight obtained there was deducted the weight of the alumina and iron oxide found by another experiment, and the difference was calculated to Na_2O. Two experiments gave:

Na_2O by titration, 78·60 and 78·65. Mean	78·63
Na_2O weighed as Na_2SO_4, 78·72 and 78·69. Mean ...	78·70
General Mean	78·66

Estimation of Alumina and Iron Oxide.

About 2 grammes were taken, dissolved in water, acidified with hydrochloric acid, and precipitated by ammonia in slight excess:

	Per Cent.
Weight of ignited ppt.	0·49

Estimation of Oxygen existing as Peroxide.

First Method tried.—From 0·2 to 0·25 gramme of the sample was weighed into a small dry tube closed at one end, which was then placed in the flask attached to a Lunge nitrometer. The flask contained a small volume of potassium permanganate solution acidified with sulphuric acid, and after the whole had acquired the temperature of the room, the mercury was levelled, and then the peroxide and the permanganate solution were brought into contact.

Weight taken in grammes.	Oxygen obtained in c.c., reduced to 0° and 760 mm. (dry).	Oxygen obtained in grammes.	Oxygen obtained per cent., assuming half came from the $KMnO_4$.
0·1974	29·78	0·0426	10·79
0·2434	37·28	0·0533	10·95
0·2457	38·56	0·0551	11·22
0·2447	38·47	0·0550	11·24

Evidently the decomposition was only partially effected in accordance with the equation:

$$5Na_2O_2 + 2KMnO_4 + 8H_2SO_4 = 5Na_2SO_4 + K_2SO_4 + 2MnSO_4 + 8H_2O + 5O_2.$$

Second Method tried.—About 0·1 gramme was dissolved in cold water previously mixed with some dilute sulphuric acid, and the solution was titrated with decinormal permanganate. Three experiments gave 18·4, 18·3, and 17·8 per cent. of oxygen.

There was a slight loss of oxygen at the moment of contact between the peroxide and the acidified water. A larger quantity, 1·0019 grammes, was dissolved in acidified water, diluted to 1 litre, and 100 c.c. titrated. Two experiments gave only 17·45 per cent. of oxygen.

Third Method tried.—An attempt to decompose the peroxide in the nitrometer with water alone was not successful, decomposition being much too slow and incomplete. But a trace of cobalt nitrate previously added to the water was found to be all that was required to effect rapid aud complete decomposition. The peroxide was weighed into the small tube, and into the flask were placed about 5 c.c. of water and one small drop of cobalt nitrate solution. Three experiments were made, and gave the following results:

Weight taken in grammes.	Oxygen obtained in c.c., reduced to 0° and 760 mm. (dry).	Oxygen obtained in grammes.	Oxygen obtained per cent.
0·2499	32·40	0·0463	18·54
0·2472	31·91	0·0456	18·46
0·2537	32·94	0·0471	18·57

The mean of these results—viz., 18·52 per cent.—was accepted. They show that decomposition by water in the nitrometer flask in the presence of a milligramme or two of cobalt sesquioxide is the most accurate, as well as the simplest, method for the assay of sodium peroxide. It would be more accurate to add precipitated cobalt sesquioxide than cobalt nitrate, as obviously a trace of oxygen is used up in oxidizing the cobaltous oxide. My result may, therefore, be a trifle too low; but, taking it as practically correct, the composition of the sample works out as follows:

Constituents found.	Per cent.	Calculated composition.	Per cent.
Na_2O	78·66	Na_2O_2	90·41
O	18·52	NaOH	8·73
Al_2O_3 and Fe_2O_3	0·49	Al_2O_3 and Fe_2O_3	0·49
			99·63

This analysis does not take into account a very little carbonate, a trace of chloride, a minute trace of sulphate, and a few particles of unoxidized sodium which the sample contained. No other impurity was detected.

Discussion.

Dr. Dupré said that the analysis of sodium peroxide was a very unsatisfactory operation to perform. All the processes he had tried were almost impossible to conduct without loss of oxygen. He had not, however, tried the cobalt process, but thought it would be a sensible improvement.

NOTE ON GLADDING'S PROCESS FOR THE DETERMINATION OF RESIN IN SOAP.

By L. Archbutt, F.I.C.

Lewkowitsch's destructive criticism of all the methods which have been proposed for the determination of resin in soaps* leaves Gladding's process in this position: that, in the absence of a really reliable process, it will continue to be used on account of its simplicity, especially in cases where the soap has been made from a known fat. I wish to direct attention to a source of error which is one cause of erroneous results.

In his original paper, "On the Quantitative Separation of Resin from Fats,"† Gladding directs that 0·5 gramme of the mixed fatty and resin acids is to be dissolved in 20 c.c. of 95 per cent. alcohol. The solution, mixed with one drop of phenolphthalein, is to be treated with a saturated alcoholic solution of caustic potash, added drop by drop until the deep-red colour characteristic of alkalinity is obtained, after which "one or two additional drops of the potash solution are added, and the flask is placed on the water-oven and kept at the temperature of boiling alcohol for ten minutes to ensure the saponification of the last traces of fat." In the analysis of soap, "one gramme of the soap in thin shavings is dissolved in the alcohol, and treated in every way as above. Particular care is paid to the saponification, as above directed, in order to make certain that no trace of unsaponified fat is left."

In my laboratory the above directions have always been carried out, but I have long been sceptical as to the value of the last part of the process, by which the absence of unsaponified fat is supposed to be ensured. On not a few occasions the resin acids extracted from soaps have been more or less soft, instead of hard and brittle. But my observations have not gone beyond this, until a few days ago, when two samples of palm soap were being tested side by side; from one the resin obtained was quite hard and brittle, and from the other soft. As the soft resin was several units per cent. higher than the hard resin, and the question under investigation related to the relative amounts of resin in the two soaps, it became necessary to make further experiments. So 10 grammes of the soap yielding the soft resin were dissolved in alcohol, and boiled down in a basin with 1 c.c. of a 50 per cent. caustic soda solution. The soap, thus thoroughly freed from unsaponified oil, was dissolved in hot water, the solution well boiled, and decomposed with dilute sulphuric acid. A suitable quantity of the washed and dried fatty and resin acids was weighed, dissolved in alcohol, neutralized with potash, and the resin acids separated as usual. They were now quite hard and brittle. The results numerically were as follow:

	Resin Acids.	
First experiment	25·18 per cent,	soft.
Second ,, ...	21·07 ,,	hard and brittle.

The crude fatty and resin acids obtained from a soap in the ordinary course of analysis would contain the unsaponified oil, and would be no better to work upon than the soap itself. The soap must, therefore, either be boiled with a sufficient

* *Soc. of Chem. Ind.*, 1893, pp. 503-508. † *Chem. News*, vol. xlv., p. 160.

excess of alkali to saponify undecomposed fat, or else the aqueous solution of the soap must be shaken with ether to remove unsaponified oil, and the acids liberated from the purified soap solution used for the resin estimation. The latter method would be preferable, as it would remove any cholesterol or other unsaponifiable matter as well as the unsaponified oil.

Discussion.

Mr. Allen said that he had found it a great improvement to precede Gladding's process by a preliminary treatment of the soda soaps of the fatty and resin acids with ether-alcohol, as directed by Barfoed. This left the greater part of the fatty acids insoluble, and it became much easier to effect a separation of the resin acids from the residual fatty acids by Gladding's silver process. But of late he, Mr. Allen, had rather preferred Twitchell's process to Gladding's, even with the preliminary Barfoed treatment. This method consisted in passing hydrochloric acid gas through a solution of the fatty and resin acids in absolute alcohol, whereby the former suffered conversion into compound ethers or esters, while the resin acids were unaffected. On diluting the product with water, and adding petroleum spirit, the resin acids and esters were readily separated from the aqueous liquid containing the hydrochloric acid, and the amount of the resin acids ascertained approximately by titrating the washed layer with standard alkali. The results so obtained could be checked by separating the lower layer, acidulating it, and weighing the liberated resin acids. There was no doubt that the exact determination of resin in soap, etc., was a very difficult problem, which could not be regarded as completely solved, and any approach to greater simplicity or accuracy would be welcomed by those accustomed to make such determinations.

Mr. Smetham said his experience of Gladding's method coincided with that of Mr. Allen. Where a large number of determinations had to be made, and something was known about the constituents of the soaps, the easiest and best plan was to compare the specific gravities of the resin and fatty acids with the specific gravities of standards made up of known quantities of resin and fatty acids from various sources, the specific gravities being taken at 100° C. In this way the percentages of resin and fatty acids could be determined with sufficient accuracy for manufacturing and commercial purposes. If the sources of the fatty acids were not known, determinations of the saponification equivalent and of the iodine absorption would, as a rule, give sufficient information to enable such cases to be dealt with.

THE COMPOSITION OF MILK, AND THE CONDITIONS AFFECTING IT, AS SHOWN BY BELL'S ANALYSES.

By J. F. Liverseege, F.I.C.

Dr. Bell's analyses of milk, dated August 15, 1893, have several times been quoted in police-court proceedings, and it seemed worth while to examine them in detail, to ascertain how far they can be quoted in opposition to this society's standard, particularly as the report accompanying them is so meagre.

The average composition is shown by the following table:

Averages.	Sp. Gr.	Solids per Cent. (Bell).			Calculated.	
		Not-Fat.	Fat.	Total.	S.-n.-F.	Fat.
273 Cows	1032·3	8·908	3·991	12·899	8·86	4·03
55 Dairies	1031·9	8·96	4·00	12·96	8·80	4·16
Dr. Vieth	—	—	—	12·90	8·8	4·1

The solids-not-fat and fat have been calculated from Hehner and Richmond's table, so that the results could be compared with Vieth's average of 120,540 samples. It will be seen that these averages are practically identical, though it is somewhat remarkable that in the dairies the calculated fat exceeds the found by ·16 per cent., while in the single cows the excess is only ·04 per cent.

The range in quality is shown by the following table. I have also calculated all the low figures of the single cows for comparison with the Society's standard.

	55 Dairies.		273 Cows.	
Total Solids.				
Under 11·5 %	0 Milks =	0 %	7 Milks =	3 %
11·5 to 11·99	4 ,,	7	37 ,,	14
12·0 ,, 12·49	6 ,,	11	69 ,,	25
12·5 ,, 12·99	19 ,,	35	44 ,,	16
13 ,, 13·99	23 ,,	42	82 ,,	30
14 ,, 14·99	3 ,,	5	22 ,,	8
15 and over	0 ,,	0	12 ,,	4
	55	100	273	100

Fat.	Bell.		Bell.		Calculated.	
Under 2·75 %	0 Milks =	0 %	6 Milks =	2 %	3 Milks =	1 %
2·76 to 2·99	1 ,,	2	12 ,,	5	10 ,,	4
3·0 ,, 3·19	1 ,,	2	21 ,,	8		
3·2 ,, 3·49	8 ,,	15	37 ,,	14		
3·5 ,, 3·99	18 ,,	32	75 ,,	27	260 ,,	95
4 ,, 4·99	26 ,,	47	95 ,,	34		
5 and over	1 ,,	2	27 ,,	10		
	55	100	273	100	273	100

Solids-not-Fat.	Bell.		Bell.		Calculated.	
Under 8 %	0 Milks =	0 %	5 Milks =	2 %	7 Milks =	3 %
8·0 to 8·29	0 ,,	0	10 ,,	4	17 ,,	7
8·3 ,, 8·49	2 ,,	4	31 ,,	11	40 ,,	15
8·5 ,, 8·69	6 ,,	11	33 ,,	12		
8·7 ,, 8·99	21 ,,	38	95 ,,	35		
9·0 ,, 9·49	25 ,,	45	71 ,,	26	209 ,,	75
9·5 and over	1 ,,	2	28 ,,	10		
	55	100	273	100	273	100

It will be seen that no dairy gave less than 11·5 per cent. of total solids, the lowest being 11·68 per cent. and 11·80 per cent., while the lowest single cows were 10·33 per cent., 11·19 per cent., 11·21 per cent., and 11·35 per cent., the other three being more than 11·4 per cent.

Among the dairies, the only one below 3 per cent. of fat is 2·89 per cent., which when calculated, gives 2·99 per cent. The two lowest single cows give 2·43 per cent., and calculate to 2·6 per cent. and 2·67 per cent. The seven milks with total solids under 11·5 per cent. have all less than 3 per cent. of fat.

Two of the dairy milks give solids-not-fat under 8·5 per cent., viz., 8·4 per cent. and 8·48 per cent.; but four of the calculated figures are below the standard, viz., 8·28 per cent., 8·33 per cent., 8·35 per cent., and 8·42 per cent. The two lowest single cows are 7·52 per cent. and 7·79 per cent., and the two lowest calculated are 7·52 per cent. and 7·66 per cent.

In all, 74 milks from single cows are below the Society's limits—10 being low in fat only, 61 in solids-not-fat only, and 3 in both; but in 37 cases there is a compensation in the other constituents, so that the total solids are 12 per cent. or over, while 26 more are within 0·2 per cent of the standard, leaving only 11 milks, or 4 per cent., that are much below the standard.

It has been stated that the coil process gives "an increase of what is called butter-fat of perhaps 0·2 per cent." over the Bell process, but it is worth while noting that sometimes the calculated fat exceeds the figures given by 0·4 per cent.; in other cases the calculated fat is lower—in one case as much as 0·26 per cent.

As in each milk, the age, food, hour of milking, and time from calving are given, as well as the breed of the cows, and the quantity of milk yielded, I tried to find if, from these analyses, any general conclusion as to the effect of these factors on the quantity of the milk could be arrived at. It might be thought that these things do not concern the members of the Society, but as the public analyst may any time be cross-examined as to the effect of food, etc., I do not think any apology is needed.

The following table gives the average composition and quantity of milk yielded by the cows of the various breeds, but it should be noticed that the number of samples in some cases is too small to be of much use.

The milk from the North Devon cows is highest in solids-not-fat and lowest in fat. The Dutch cows give lowest total solids and solids-not-fat, while the Jersey milks are highest both in total solids and in fat. The cross-breeds give the greatest, and the Kerries the least yield of milk:

Breed.	Number of Cows.	Solids-not-Fat.	Fat.	Total Solids.	Average Yield. Quarts.
Sussex	2	8·92	3·39	12·31	7·5
Welsh	2	9·15	4·40	13·55	4·2
Guernsey	4	9·30	5·16	14·46	4·1
Jersey	6	9·22	5·43	14·65	3·7
Kerry	5	8·87	4·67	13·54	2·7
North Devon ...	6	9·68	3·43	13·11	3·8
Dutch	13	8·65	3·75	12·40	5·3
Ayrshire	15	9·22	4·24	13·46	4·6
Cross-breeds ...	17	9.01	3·99	13·00	5·6
Shorthorns	203	8·86	3·92	12·78	5·3
General Average ...	273	8·908	3·991	12·899	

As there was such a difference in the various breeds, it was deemed advisable to eliminate this factor and investigate the shorthorns only. It is well known that morning milk is poorer than evening, and it has been suggested that this is due to the difference of time elapsing between milking. To test this, the facts with regard to each cow were copied on a strip of cardboard, and the strips sorted. For solids-not-fat they were divided into three classes—under 8·5 per cent., 8·5 per cent. to 9 per cent., and 9 per cent. and over—and each class subdivided into first meal and second meal, and the numbers in each class counted and calculated to percentages. Thus, 23 per cent. of the milks under 8·5 were first meal, and 77 per cent. second meal. The fats were similarly classified, and then both were arranged with regard to hours from last milking.

Figures on the same horizontal line may be compared, but vertically each class adds up to 100:

	Solids-not-Fat.			Fat.			Average Quantity. Quarts.
	Under 8·5.	8·5 to 8·99.	9·0 & over.	Under 3·5.	3·5 to 4·49.	4·5 & over.	
First meal ...	23	29	29	55	21	5*	
Second meal ...	77	71	71	45	79	95	
Hours between milking:							
8 to 9 hours	30	29	30	11	28	60	4·4
10 ,, 11 ,,	48	41	37	29	50	36	5·1
12 ,, 13 ,,	17	16	16	24	17	2*	6·4
14 ,, 16 ,,	5*	14	17	36	5	2*	5·9

This table shows that while 55 per cent. of the milks low in fat were first meal, only 5 per cent. of those rich in fat belong to this meal. Also that, while 11 per cent. of poor milk were 8 to 9 hours between milking, 60 per cent. of the milks high in fat were in this class, showing that the longer the time between milking, the larger will be the proportion of milks low in fat, a conclusion which is supported by the other figures. If, however, the solids-not-fat be examined, it will be seen that time makes practically no difference, but a longer interval has a slight tendency to decrease the proportion of poor milks.

This shows that for the study of the effect of food on the amount of fat, etc., it is necessary to select either first or second meal cows, or the differences due to the meal would mask the smaller variations; the second-meal class, being the larger (147 cows), was selected, but all shorthorns were taken for the solids-not-fat:

* Number of cows small.

	Solids-not-Fat.			Fat.		
	Under 8·5.	8·5 to 8·99.	9·0 & over.	Under 3·5.	3·5 to 4·49.	4·5 & over.
Age 2 to 4½ years	11*	34	32	31	33	24
5 ,, 6 ,,	62	36	46	54	39	65
7 ,, 8 ,,	16	24	22	11*	19	11
9 ,, 12 ,,	11	6	0	4*	9	0
Yield 1 to 3½ quarts	25	23	28	19	22	37
4 ,, 5 ,,	45	27	31	39	41	30
6 ,, 7 ,,	17	37	27	23	30	30
8 ,, 10 ,,	13	13	14	19	7	3
Time from Calving: 1 to 6 weeks	18	28	30	15*	28	27
7 ,, 12 ,,	37	36	16	38	33	25
3½ ,, 6 months	27	23	30	31	28	18
7 ,, 9 ,,	10*	8	16	8*	6	27
10 months & over	8*	5	8	8*	5*	3*
Percentage of cows fed on						
Hay	20	48	32	17	55	28
Mangolds or Swedes ...	22	47	31	16	55	29
Brewers' or distillers' grains	21	43	36	14	50	36
Linseed or cotton-seed cake	25	45	30	16	62	22
Chaff or straw	25	49	26	17	64	19
Meals (oat, pea, barley, etc.)	18	53	29	19	62	19
Grass	15	56	29	19	56	25

With regard to age, it may be noted that cows five and six years old give a large proportion of the milks, and that no milks high in fat or solids-not-fat occur in milk from cows over eight years old.

When the quantity of milk is under one gallon, the proportion of milks high in fat is increased, and when two gallons and over is yielded at a milking, there is a small proportion of rich milks. The milks given by cows for periods of less than seven weeks after calving have a small proportion poor both in fat and solids-not-fat, and those seven to nine months after calving are of a better quality, particularly in fat.

With regard to the effects of food, the problem is very complicated, as the majority of cows are fed on from three to six different kinds. The figures given are the percentage of rich, medium, and poor milks yielded by cows fed on each food considered by itself—thus, out of 100 cows fed on hay, 20 gave milk that was low, 48 medium milk, and 32 milk high in solids-not-fat. Some of the cows were fed on grass only, and with regard to part of them it is remarked that the grass was very scarce. The very small variations appear to show that food by itself has not much effect on the composition of the milk if the cows are fairly fed, as these are supposed to be. The large proportion of good milks from cows fed on grains is notable.

* Number of cows small.

The one respect in which the information given about these milks is deficient, is that no date is stated, and as the time of the year exercises a marked effect on the composition of the milk, one feels that, as this could not be allowed for, the influence of season may have introduced some errors into the above results. To summarize what has been said, these analyses show

(1) That, with four very slight exceptions, all the mixed milks are up to the Society's standard.

(2) That only 4 per cent. of the milks from the single cows would show signs of much adulteration, as judged by the Society's standard, if allowance is made for excess of the other constituents.

(3) That analyses by the Bell method may give from 0·40 less to 0·26 per cent. more than the calculated figures.

(4) That the longer the time between milking, the lower will be the fat in milk.

(5) That, with the exception of breed, the other conditions have much less effect on the proportion of poor milks.

ON ROASTED CHICORY.

By E. G. Clayton.

(*Read at the Meeting, October* 29, 1894.)

The amount of sugar present in roasted chicory is very variously stated; and in most cases, apparently, only the sugar before inversion has been estimated. The following figures, compiled from Allen's "Commercial Organic Analysis" and other sources, represent the quantities of sugar mentioned by different observers; and some other numbers are quoted in the table for comparison:

Table I.

	Sugar before inversion (Glucose).	Sugar after inversion (expressed as Sucrose?).	Total Sugar.	Proteids.	Water.	Mineral Matter.
Petermann *a.* Coarse grains.	26·12	—	—	6·38	16·28	7·16
Petermann *b.* Fine powder.	23·76	—	—	6·64	16·96	8·44
Krauth*	22·39	21·18	43·57	—	4·30	10·36
Hassall *a.*	12·2	—	—	—	14·5	4·3
Hassall *b.*	10·4	—	—	—	12·8	6·8
König†	15·87	—	—	6·09	12·16	6·12
Graham, Stenhouse, and Campbell *a.* ...	11·98	—	—	—	—	—
Graham, Stenhouse, and Campbell *b.* ...	15·96	—	—	—	—	—
Graham, Stenhouse, and Campbell *c.* ...	17·98	—	—	—	—	—
Graham, Stenhouse, and Campbell *d.* ...	9·86	—	—	—	—	—

* Krauth's figures (except for moisture) originally referred to the substance dried at 100°, but for the above table they have been re-calculated.

† The number obtained by König, given in column 1, is merely stated to be "sugar," and almost certainly refers to glucose only.

Had the proportions of sugar after inversion been also determined, it is probable that very considerable numbers would have replaced the blanks in columns 2 and 3; and in the light of some results about to be summarized, certain of the figures in column 1 are so high as to suggest the possibility that some of the samples contained added sugar. This is further indicated by the low proteids (column 4).

Specimens of whole chicory root and (for comparison) dandelion root were carefully roasted and ground in the writer's laboratory, and extracted with boiling water. The solutions were decolorized by treatment with lead acetate, filtered after the addition of sodium carbonate, and titrated, before and after inversion, with Fehling's solution. A sample of commercially-roasted and ground chicory, purchased as pure, was similarly treated, with the exception that hot alcohol was in this case used, in place of water, for the extraction. The proportions of mineral matter were determined in the two laboratory samples, and are here stated, together with the other results:

TABLE II.

	Matter reducing Fehling's solution before inversion (expressed as Glucose).	Matter reducing Fehling's solution after inversion (expressed as Glucose).	Matter reducing Fehling's solution after inversion (expressed as Sucrose).	Total (*i.e.*, the figures in columns 1 and 3), expressed as Glucose + Sucrose.	Mineral matter.
Chicory root (roasted in laboratory) ...	5·66	14·34	13·62	19·28	5·18
Dandelion root (roasted in laboratory) ...	5·66	20·43	19·41	25·07	4·05
Chicory root (commercially roasted and ground) ...	7·19	21·58	20·50	27·69	—

It will be seen that the numbers for sugar before inversion (column 1) are considerably lower than the corresponding figures in the analyses previously cited.

Some samples of roasted chicory, undoubtedly containing added saccharine matter, lately came into the writer's hands, and yielded the following results on partial analysis:

TABLE III.

	Matter reducing Fehling's solution before inversion (expressed as Glucose).	Matter reducing Fehling's solution after inversion (expressed as Glucose).	Matter reducing Fehling's solution after inversion (expressed as Sucrose).	Total (*i.e.*, the figures in cols. 1 and 3), expressed as Glucose + Sucrose.	Proteids.	Water.	Mineral matter.
Sample 1 (coarse grains or nibs) ...	16·66	46·50	44·17	60·83	—	—	—
Sample 2 (powder)	12·00	62·50	59·37	71·87	—	—	—
Sample 3 (coarse grains or nibs) ...	13·53	33·75	32·06	45·59	8·19	—	—
Sample 4 (powder)	9·24	23·61	22·43	31·67	9·31	1·71	6·25

It is obvious that these samples contained large proportions of foreign saccharine matter, if the number in Table II., representing the amount of sugar expressed as sucrose in the laboratory-roasted chicory root, be provisionally taken as a basis of calculation. The unusually small percentage of water found in the fourth sample is noteworthy; and it will be observed also that, in spite of the presence of considerable amounts of sugar, the quantities of proteids in samples 3 and 4 are larger than the proportions quoted in Table I.—already alluded to as being markedly low.

The intensity of the colour of the infusions led to some experiments as to the most suitable methods of extraction and decolorization; a series of estimations of glucose and invert sugar being made, after various means of removing the colour had been employed. An opportunity, of which the writer gladly avails himself, here arises of acknowledging the very careful assistance in these sugar determinations willingly rendered him by Mr. Sidney B. Wright.

The results obtained are collated in Table IV.

TABLE IV.

	Infusion decolorized by	Sample 1 (Grains). Sugar, expressed as Glucose.		Sample 2 (Powder). Sugar, expressed as Glucose.		Sample 3 (Grains). Sugar, expressed as Glucose.		Sample 4 (Powder). Sugar, expressed as Glucose.	
		Before inversion.	After inversion.	Before inversion.	After inversion.	Before inversion.	After inversion.	Before inversion.	After inversion.
Extracted with water.	Lead acetate only	16·66	46·50	12·00	62·50	—	—	—	—
Extracted with alcohol.	Lead acetate only	15·79	44·21	12·50	62·50	13·53	33·75	9·24	23·61
	Lead acetate, followed by filtration through animal charcoal	15·00	43·07	—	—	—	—	—	—
	Milk of lime only	7·14	47·40	—	—	—	—	—	—
	Lead acetate and milk of lime together	0	14·28	—	—	—	—	—	—
	Lead acetate and milk of lime, followed by filtration through animal charcoal	—	—	—	—	2·11	24·28	0	18·75

From the above results of duplicate determinations in samples 1 and 2, it is clear that the difference, caused by using alcohol in place of water for the extraction of the sugar, is inconsiderable. As for the several processes used to decolorize the solutions, the results in the first of the four main columns of the table (all of which results were yielded by the same sample, No. 1, and are in this respect strictly comparable), show that filtration through animal charcoal, though removing some

colouring matter which lead acetate had failed to precipitate, effected this at the expense of a small but perceptible reduction in the percentages of sugar. Milk of lime, alone, yielded a pale-tinted solution, but removed nearly one-half of the "sugar before inversion." The addition of lead acetate, followed immediately by milk of lime, resulted in a nearly colourless solution; but practically all the glucose, and more than two-thirds of the "sugar after inversion," had vanished. Confirmation of these results appeared in the numbers yielded by samples 3 and 4 (columns 3 and 4): after the use of lead acetate and milk of lime, followed by filtration through animal charcoal, far lower results were obtained than when lead acetate only had been employed. In short, the use of milk of lime, alone or in conjunction with lead acetate, is quite inadmissible, as, perhaps, might have been anticipated; and the process of extraction with hot water, followed by simple decolorization with lead acetate, appears most suitable in the case of chicory. Filtration through animal charcoal may, in addition, be necessary in many instances, and the influence on the final results will not be very serious.

Addendum.

Since the above was written I have read the evidence on chicory given before the Food Products Committee, and I venture to quote a portion of it here.

After Mr. Bannister's remark, that "there is a greater extractive got from chicory now than there used to be ten or fifteen years ago," questions 866 and 867 followed:

"866. Is that agreed among chemists, or is it owing to your improved methods of analysis?—It is on account of the *difference of growth*, I believe, and not to improved methods of analysis.

"867. That the quality of chicory has improved?—It contains more extractive matter, and is *richer in sugar*." [Italics are mine.]

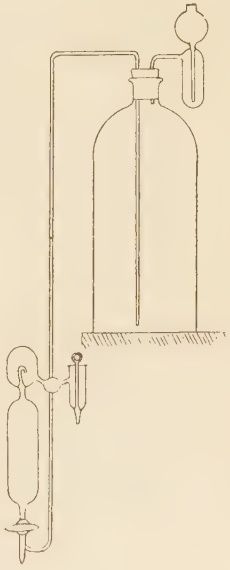
If roasted chicory be referred to (and this is presumably the case), may not the explanation of the greater richness in sugar and extractive matter be found, *not* in any "difference of growth," but in a frequent practice of such extraneous addition as that described in the above paper?

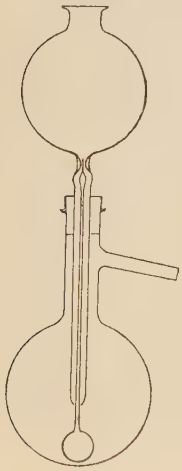
APPARATUS FOR PRESERVING AND DELIVERING STANDARD SOLUTIONS.

By J. C. Chorley.

(*Read at the Meeting, November* 7, 1894.)

This apparatus will be found useful for preserving and delivering known volumes of such solutions as alcoholic potash, which are liable to contamination by exposure to air. The wash-bottle inserted in the large store-bottle is filled with a solution of caustic potash, and so also are the bulbs connected with the top of the pipette. By means of the two-way cock, this can be filled to overflowing, and a known quantity of the solution can be delivered by running off to a mark on the narrow part of the pipette.





CONSTANT-LEVEL APPARATUS.

By J. C. Chorley.

(*Read at the Meeting, November* 7, 1894.)

This apparatus was designed for use in distillations where it is advisable to maintain a constant level of liquid in the distillation-flask. The small bulb at the end of the long thin tube floats on the surface of the liquid. Its upper end, which is ground, fits into the thickened part of the larger tube, and prevents the water contained in the large bulb from flowing into the flask. When, however, the level of the liquid falls, the upper end of the long thin tube drops away, and releases a small quantity of water. In this way the level is automatically maintained.

The Composition of Honey-dew and the Influence of a Summer rich in Honey-dew on the Condition of Honey. Dr. Ed. von Raumer. (*Zeit. für Anal. Chemie*, 1894, pp. 397-408).—Honey-dew obtained from maple leaves by solution in cold water, and subsequently decolorized by animal charcoal, had the following percentage composition:

	I.	II.
Dry substance	75·12	84·08
Water	24·88	15·92
Sugar in 100 c.c. dry substance direct	16·70	17·70
,, ,, ,, inverted	28·5	28·60
Polarization 10 grammes dry substance direct	23·2°	22·90°
,, ,, ,, ,, inverted	21·4°	20·80°
Dextrin after fermentation with yeast	39·4	—
Ash	3·02	2·86
Nitrogenous matter calculated as albumen	3·17	—
In the ash { CaO	19·5	—
In the ash { SO_3	16·2	—

The sugar was estimated by the Soxhlet-Allihn method, and the quantity compared with that obtained by fermenting the honey with yeast, and calculating the amount of sugar from the yield of alcohol. This gave 10·7 per cent. more sugar than could be obtained by direct estimation. The author's explanation is that there is an intermediate product between sugar and dextrin which is capable of fermentation, but which does not readily reduce Fehling's solution. The method was also used as a check on the estimation of sugar in honey.

From experiments with honey produced in 1893—a year noticeably rich in honey-dew—the following conclusions are arrived at:

1. All species of honey contain dextrin fermentable with difficulty. Some, in particular those derived from conifers, contain considerable quantities of unfermentable dextrin. Omitting the abnormal case of conifer honey, this large quantity can

only be explained on the supposition that the honey has been mixed by the bees with honey-dew. An increase in the normal amount of mineral constituents and nitrogenous matter may also point to the same conclusion.

2. Direct dialysis of the honey by Hänle's method* is not advisable, since even after 40 to 50 hours sugar is left on the membrane; and on the other hand dextrin has passed through. The difficulty is obviated by dialysing the fermentation products instead of the fresh honey.

3. The dextrins of honey-dew probably undergo some change in the honey-bag of the bee, so that their power of dialysis is increased. C. A. M.

Zinc-bearing Spring Waters. W. F. Hillebrand. (*Bulletin U.S. Geological Survey*, No. 113. *Report of Chemical Work in* 1891-92 *and* 1892-93).—Analyses are given of two spring waters from Missouri, both containing zinc sulphate as a natural constituent. One contained 120·5 parts of Zn and 61·1 of Ca per million. The other 132·4 of Zn and 63·1 of Ca. A peculiarity in the waters was that, although some of the Ca existed as carbonate, this being shown by the excess of bases over Cl and SO_3, yet *all* the CO_2 was expelled by simply boiling the water. The amount of CO_2 obtained was 120·5 per million in the first water, and 110·0 per million in the second, and no more could be obtained by acidifying the waters. The explanation is that the $CaCO_3$ at a temperature near the boiling-point of the water reacts with the $ZnSO_4$, forming $CaSO_4$, zinc hydrate or a very basic sulphate, and CO_2. A determination of the residue, by simply evaporating the water to dryness would therefore involve a serious error.

C. A. M.

On the Estimation of Phosphoric Acid by titration of the Molybdic Precipitate. B. W. Kilgore. (*Jour. Amer. Chem. Soc.*, 1894, pp. 765-772.) — A summary of different processes is given. The author obtained good results in general by Pemberton's (Analyst xix., p. 135) method, though high ones were not uncommon. The variations were found to be due to deposition of molybdic acid from the molybdate solution. By precipitation at a temperature of from 55° to 60° instead of at 100°, and by the use of the official molybdate solution of the Association of Official Agricultural Chemists instead of the more dilute solution used by Pemberton, this error may be avoided. The results tabulated agree well with those obtained by gravimetric determination. The official molybdate solution is prepared by dissolving 100 grammes of molybdic acid in 417 c.c. of ammonia (sp. gr. 0·96) and pouring this into 1250 c.c. of HNO_3 (sp. gr. 1·20). C. A. M.

A Comparison of Methods for the Determination of Starch. W. E. Stone. (*Jour. Amer. Chem. Soc.*, 1894, xvi., No. 11, pp. 726-733.) — A comparative examination of different methods of starch analysis proves that, while the ordinary methods give excellent results with pure starch, the results obtained with substances

* *Zeit. für Anal. Chem.*, 33-99.

containing other carbohydrates are discordant. This is due to the pentosans or gums acting towards some reagents precisely like starch.

The table gives the results obtained by the following five methods.

1. *Sachsse's Method.*—From 3 to 5 grammes of the material are heated in the water-bath for three hours with 200 c.c. of water and 20 c.c. of concentrated HCl. The glucose formed is estimated by Fehling's solution. According to Märker, this method gives too low results, owing to destruction of glucose by the long heating with the acid.

2. *Guichard's Method.*—Three grammes of the material are heated in the water-bath for one hour with 100 c.c. 10 per cent. nitric acid, made up to 200 c.c. and polarized. The readings are calculated to glucose.

3. Three grammes of the material are heated in the water-bath with 100 c.c. of saturated solution of oxalic acid for an hour, in order to bring the starch into solution. The liquid is cooled, made up to 200 c.c. with 10 per cent. nitric acid, and heated in the water-bath for another hour. The solution is then polarized.

4. *Baudry's Method.*—A normal weight, 5·376, is adopted for the Laurent polarimeter. This weight, or some multiple of it, is placed in a 200 c.c. flask, with 100 c.c. of water, 0·4 to 0·5 of salicylic acid added, and heated in the water-bath for half an hour. The contents of the flask are made up to within 20 c.c. of the mark with hot water, rapidly cooled, a few drops of ammonia added to clarify, filled up to the mark, filtered, and polarized in a 400 mm. tube.

5. *Asboth's Method.*—See ANALYST, vol. xii., p. 138.

Materials.	Inversion by HCl.	Inversion by HNO_3.	Inversion by oxalic acid and HNO_3.	Solution by salicylic acid.	Precipitation by $Ba(OH)_2$.
1. Potato starch	85·75	85·5	85·75	85·47	85·58
2. Dried potato	70·92	69·79	68·53	—	64·25
3. Wheat flour	77·69	70·65	65·29	69·38	59·76
4. Corn meal	73·24	66·81	70·55	—	62·11
5. Wheat bran	65·86	40·25	38·68	—	70·77
6. Hay...	3·48	19·10	19·10	—	66·47
7. Wheat middlings	30·00	63·09	60·24	—	60·44
8. Cotton-seed meal	4·15	—*	—*	—	54·65
9. Mixture of starch, sugar, and dextrin	9·58	21·00	24·08	18·8	33·99

The potato-starch, but for moisture and cellulose, was "pure." Each mode gave credit for the entire amount of real starch present.

The following experiment proves that the pentosans behave like starch. Xylan, free from other carbohydrate, was obtained from wheat straw. One gramme was heated for thirty minutes with 50 c.c. of water and 4 c.c. of strong HCl. The neutralized solution was made up to 100 c.c. Of this, 6·7 c.c. were required to reduce 10 c.c. of Fehling's solution, a quantity equivalent to 67·16 per cent. of starch. The other methods gave similar results, with the exception of the salicylic acid method, which was found not to be applicable.

* These solutions were lævo-rotary.

The author, therefore, concludes that no method based on the foregoing principles is suitable for accurate work on grains or forage material. He suggests the use of some method by which the starch may be brought into solution without other carbohydrates, *e.g.*, by means of diastase or malt infusion. The weighed material, after being made into a paste with boiling water, is cooled to 65°, a small quantity of malt infusion added, and the temperature maintained at from 60° to 65° for half an hour. After the starch reaction has disappeared, the solution is filtered, the residue well washed, and the filtrate, after warming with a few c.c. of acid and then neutralizing, is made up to definite volume and titrated. The preliminary experiments cited indicate that the pentosans and similar bodies are not affected by diastase. C. A. M.

A Method for Determining Calcium Oxide in Quicklime. W. E. Stone and F. C. Scheuch. (*Jour. Amer. Chem. Soc.*, 1894, xvi., No. 11, pp. 721-725.)—Since the value of quicklime depends on the quantity of CaO present, a method for determining this is proposed, which is based on the fact that the alkaline earths form definite compounds with sucrose. With CaO at least three are known, containing respectively one, two, and three molecules of CaO to one of sucrose, the two first being formed by dissolving CaO in the cold in a sucrose solution, while the third is precipitated on warming the solution.

The preliminary experiments cited confirm Lamy's* observation that the solubility of CaO in sucrose solution varies inversely as the temperature, and prove that not less than 150 c.c. of a 10 per cent. sucrose solution are required to dissolve the CaO in one gramme of quicklime. Other ordinary constituents of quicklime, such as iron, alumina, magnesia, silica, and calcium carbonate are either quite insoluble in the sucrose solution, or soluble only to an inappreciable extent. One gramme of magnesia, after being shaken with 150 c.c. of sucrose solution, showed only 0·001 gramme of MgO in the clear filtrate.

The following is the method adopted: One gramme of the finely-powdered substance is shaken for twenty minutes with 150 c.c. of 10 per cent. sucrose solution, the liquid filtered, and the clear solution titrated with standard HCl, half an hour being sufficient for the whole operation. Test samples of quicklime, containing very little $CaCO_3$, were analysed by this method, and the results compared with those obtained in the usual way by precipitation as calcium oxalate.

Sample.	Quicklime taken. Grammes.	Volume of 10 per cent. sucrose solution taken.	Per cent. CaO by titration method.	Per cent. CaO by gravimetric method.	Variation of volumetric method.
1.	1·020	150	92·12	93·00	—0·88
2.	1·090	150	91·90	92·28	—0·38
3.	1·006	150	92·15	93·10	—0·95
4.	1·108	150	95·01	95·90	—0·89
5.	1·023	150	87·30	87·70	—0·40
6.	1·232	150	91·70	92·30	—0·60

The small quantity of $CaCO_3$ present would account for the discrepancy between the gravimetric and the sucrose methods. C. A. M.

* *La sucrerie indigene et coloniale*, 11-19.

Method for obtaining Pure White Magnesium Pyrophosphate in the Citrate Method. Dr. Hugo Neubauer. (*Zeit. für angewand. Chem.*, 1894, Heft 22, pp. 678, 679.)

In the estimation of phosphoric acid by the citrate method, it is often almost impossible to obtain the precipitate perfectly white even after long-continued ignition. This may be remedied by the addition of ammonium sulphate (5 grammes) before precipitating with the magnesia mixture. When heated for five minutes over a strong flame, the precipitate becomes perfectly white. Any precipitate of calcium sulphate formed by adding the ammonium sulphate may be ignored, since $CaSO_4$ is readily soluble in ammonium citrate.

C. A. M.

A New Method of Estimating Milk-fat. Dr. P. Fernandez-Krug and Dr. W. Hampe. (*Zeit. für angewand. Chemie*, 1894, Heft 22, pp. 683-687.)—In this process a measured volume of the milk is mixed with a finely-divided mineral substance, and the water removed by adding another finely-powdered material which combines with it without affecting the milk-fat. The dried mass is shaken out with ether, and the dissolved fat estimated by evaporation of a definite portion of the solution.

The following are the details of the actual estimation: About 7·5 grammes of washed and dried kaolin are introduced into a nickel basin holding about 200 c.c., and made into a creamy paste with 5 c.c. of the milk. This is then well mixed with about 5 grammes of finely-powdered anhydrous sodium sulphate, which combines with the water in the milk. A fine state of division is necessary to prevent a crystallization of the sulphate with the water, which would cause a loss of milk-fat. The dried mass is placed in a flask holding about 100 c.c. and closed with a well-fitting stopper, and 25 c.c. of ether added. After shaking for five minutes and cooling to the temperature of the air, 5 c.c. of the clear liquid are removed by means of a pipette, the point of which is covered with cotton-wool to prevent particles entering, and the dissolved fat estimated by evaporation in a weighed flask. From this the percentage on the whole can be calculated. The results obtained by this method agree well with those obtained by the older method, in which sand is mixed with the milk and the fat subsequently extracted with ether. Results in grammes per litre:

	Full Milk.	Full Milk.	Watered Milk.	Skim Milk.	Skim Milk.
Old Method ...	34·6	36·0	21·2	2·7	3·2
New Method ...	34·6	36·0	21·2	2·9	3·3

When analysing butter-milk or sour milk it is necessary to have them as homogeneous fluids. To effect this, a litre of the butter-milk or sour milk is warmed to about 45°, shaken for five minutes with 10 grammes of $NaHCO_3$, and allowed to cool to the temperature of the room. The authors promise their results as soon as they have proved that, besides fat, no other constituents of the neutralized milk dissolve in ether.

The contribution concludes with a description of an apparatus by means of which,

from the weight of the fat, its percentage in the milk can be immediately read off. It is on the principle of the steel-yard with a running weight, the mass of which is calculated for the specific gravity of the milk and other conditions. C. A. M.

On the Estimation of Nitrogen in Nitro-cellulose. Duschan Stanojewitsch. (*Zeit. für angewand. Chemie*, 1894, Heft 22, pp. 676-678.)—Lunge's method of estimation by measurement of the gas liberated by the action of H_2SO_4 and mercury on the substance is open to the objection that the action of the air in the flask on the nitric oxide causes an error. To avoid this, the author has devised an apparatus which combines the methods of Schloesing and Lunge.

The weighed substance is introduced, together with 50 or 60 grammes of mercury, into the decomposition flask, which is connected with a modified nitrometer filled with a 25 per cent. solution of soda. After the air in the flask has been displaced by a current of CO_2, 10 to 15 c.c. of H_2SO_4 are introduced, and the flask shaken until all the gas is liberated. To completely drive it over into the nitrometer, the flask is warmed while a current of CO_2 is passed in. For measurement it is advisable to introduce the gas into a measuring cylinder filled with water, which can be done by means of a delivery tube with a stop-cock at the top of the nitrometer. The apparatus is especially suitable for the estimation of nitro-glycerine in dynamite. Minute pieces of the sample are digested in the flask with H_2SO_4 and mercury. The decomposition is rapid, and the presence of carbonates does not influence the result. In the case of gelatinized nitro-cellulose it is necessary to warm the mixture over a water-bath, but the mercury and nitro-cellulose should not be warmed before the introduction of the H_2SO_4, since the reaction is then so violent that an explosion may occur. C. A. M.

A New Method of Estimating Glucose by Alkaline Copper Solution. MM. Allein and Gaud. (*Jour. Pharm. et Chim.*, 1894, xxx., pp. 305-307.)—To avoid errors introduced by the base keeping the copper in solution, the authors recommend the substitution of ammonia for the fixed alkalies. To cause ammonia to act on glucose it must be kept in a sealed tube with it at 100° for thirty or forty hours, and even then only a small quantity of the glucose is destroyed, 5 to 6 per cent. of formic acid being produced; whereas glucose, treated in a similar way with the fixed alkalies, yields up to 60 per cent. of lactic acid.

The solution is prepared by dissolving 8·7916 grammes of electrolytic copper in 93 grammes of H_2SO_4, diluting with an equal quantity of water and making up to a litre with concentrated ammonia solution. 10 c.c. of this is equivalent to 0·05 gramme of glucose. Being free from organic impurities, the solution keeps well.

In the estimation, 10 c.c. of the copper solution are measured into a flask and 10 c.c. of ammonia solution added. The flask is furnished with a cork with three holes, so that the extremity of the burette containing the sugar solution can be inserted and a current of hydrogen introduced. It is placed on a water-bath maintained at a temperature of from 90° to 95° while the hydrogen is passed in. When the temperature

of the interior of the flask is at 80°, the sugar solution is introduced drop by drop from the burette until the copper solution is decolorized. Cuprous oxide is formed, but is dissolved to a colourless liquid at the moment of formation by the ammonia, so that it is easy to judge when the blue colour is about to disappear. Excellent results are said to be obtained by the process, the only disadvantage of which is the necessity of manipulating at from 80° to 85° in order not to drive off the ammonia.

C. A. M.

On the Estimation of Boron. Henri Moissan. (*Bull. Soc. Chim. de Paris.* 1894, 18-19, pp. 955-958.)—The process is a modification of Gooch's method, by which the estimation is made more rapid and loss by volatilization avoided.

The weighed boron compound, which must be in the form of boric acid or a borate, is placed in a distilling tube, the neck of which is closed by a stoppered funnel ground into it. One c.c. of HNO_3 is introduced, and the contents of the flask distilled to dryness by means of a water-bath. The distillate, condensed by the usual spiral worm, falls into a flask containing an excess of ammonia and connected with bulb tubes also containing ammonia. The residue in the flask is distilled with successive portions (10 c.c.) of pure methyl alcohol, being taken to dryness each time, until the alcohol distils over free from boric acid, as shown by the flame test. The ammoniacal distillate is emptied into a platinum basin, in which is a known quantity of calcium hydrate, previously weighed in the anhydrous state. On evaporation over a water-bath at about 70°, borate of calcium is left, and the increase of weight gives the quantity of boric anhydride from which the boron in the sample can be calculated. The calcium oxide is prepared by calcining pure calcium nitrate. A large excess is necessary : for 0·5 grammes boric acid, about 8 or 10 grammes.

The method gave the following results when used to estimate boric anhydride prepared from a pure boric acid :

I.	II.	III.	IV.	
99·73	99·67	99·62	99·6	per cent.

The author has used the process for the estimation of boron in the phosphate and sulphate of boron and in metallic borates and borides. C. A. M.

Toxicological detection and estimation of Arsenic. Ernest Barillot. (*Bull. de la Soc. Chim. de Paris,* 1894, Tomes xi. and xii., pp. 958, 959.)—Marsh's process is modified so that the arsenic deposited in the tube may be weighed. The generating flask is connected with a tube of green glass heated in three sections by burners. The substance to be analysed, after the animal matter has been destroyed by one of the ordinary processes, is treated with fuming nitric acid and potassium nitrate, the nitrous acids being afterwards removed by heating with excess of sulphuric acid. The solution is concentrated down to about 200 c.c., and after being proved to be free from nitric and nitrous acids, is introduced gradually into the generating flask, where the gas is liberated in the usual manner. The sections in which the rings of arsenic are deposited are detached and weighed, washed with fuming HNO_3, dried and again weighed, the difference giving the amount of arsenic. C. A. M.

Separation of Copper from Cadmium, Zinc and Nickel. P. N. Rikowa. (*Chem. Zeit.*, 1894, xviii., 1739.)—The author utilizes the reaction given by a soluble iodide and phenylhydrazine with a copper salt (see preceding abstract) to separate copper from cadmium, zinc and nickel. The solution containing the metals is acidulated with sulphuric acid, treated with phenylhydrazine (the hydrochloride may be used, as the presence of a chloride is of no significance in this use of the reaction) and precipitated with potassium iodide, added drop by drop, to avoid an excess, in which the precipitate is somewhat soluble. Provided enough phenylhydrazine be present, a small excess of potassium iodide is not detrimental. The copper is completely separated, and the remaining metals are retained in solution. The remaining stages of the determination may be performed by the ordinary methods. Salts of mercury and iron must be absent, as they yield precipitates with phenylhydrazine.

B. B.

The Purification of Saltpetre from Perchlorate. V. Panaotović. (*Chem. Zeit.*, 1894, xviii., 1567.)—The author confirms the statements of previous observers that commercial saltpetre is usually contaminated with perchlorate (*vide* The Analyst, xix. 22). Out of 180 casks from a well-known Hamburg firm, 122 contained more than 0·25 per cent. of perchlorate, and only 7 were perfectly free therefrom. Although saltpetre containing perchlorate must have been used for years in powder manufacture, yet it does not necessarily follow that the presence of this impurity is a matter of indifference. Explosions that have received no satisfactory explanation may have been due to it, the more so as pure potassium perchlorate, when substituted for potassium chlorate in the manufacture of signal-lights, has proved unstable, spontaneous ignition taking place after four months' storage. These facts are sufficient to render it desirable to remove perchlorate from saltpetre intended for the manufacture of explosives. An attempt to effect this by fusing the salt until the perchlorate had been decomposed proved a failure, as much nitrite was formed, together with some free alkali, the removal of which products was difficult and costly. The purification was eventually achieved by dissolving the saltpetre in hot filtered spring-water (free from chlorine) to a strength of 48° B, adding a little size to favour the separation of insoluble impurities, decanting and allowing the solution to cool to 25° C, stirring to promote the separation of small crystals. The mother liquor (28° B) was poured off, and the crystals washed five or six times with pure water. The mother liquor was concentrated, and the separated crystals re-treated. From the final mother liquor crystals of potassium perchlorate were isolated and identified.

B. B.

Preservation of Starch Solution. H. Král. (*Pharm. C. H.*, 1894, xv., 606; through *Chem. Zeit.*)—The author recommends that the starch solution used in carrying out Hübl's iodine absorption process be prevented from decomposing by the addition of chloroform. A small quantity of chloroform is added, and the mixture well shaken. The product can be kept for months without suffering change.

B. B.

Detection of Nitrites in presence of Iron Salts. H. Král. (*Pharm. C. H.*, 1894, xv., 619.)—The detection of nitrites by means of an iodide and starch solution —*e.g.*, in drinking-waters—fails in the presence of ferric salts, as the latter also liberate iodine. The acid necessarily added is a source of error, even when acetic acid is used, as iodine is slowly liberated in the absence of nitrites. These drawbacks can be avoided by the use of oxalic acid, in the presence of which ferric salts are stated to liberate no iodine. B. B.

REVIEW.

The Sale of Food and Drugs Acts. Bell and Scrivener. Second edition. (Shaw and Sons, Fetter Lane, E.C. Price 4s. 6d.)

The appearance of a second edition of this book affords additional evidence of the increased interest taken in the working of the Food Acts. The chemical reviewer would be guilty of a presumptuous act were he to venture upon a criticism of the purely legal portions of a book written by two learned barristers, but as far as we can see, the authors have recorded, in a very systematic manner, the most important of the numerous decisions given in courts of law touching the Sale of Food and Drugs Acts. Perhaps we might have been spared a good many of the decisions of the lower courts, which, as every analyst knows, are puzzling and contradictory.

But the authors have attempted to do more than write a legal guide, for, apparently without any chemical qualification, they have ventured to enter into the discussion of matters purely chemical, with results such as might have been anticipated. In commenting upon Section 6, truly described as "a most important section, and the entire working of the Sale of Food and Drugs Act very nearly depends upon it," the authors discuss the composition of the more important articles of food. We are sorry to find that the remarks under this head are neither characterized by the absence of bias nor by the presence of modern knowledge. They often appear to be a faint echo of the information contained in a well-known little handbook on "The Chemistry of Foods" published eleven years ago. Thus, the remarks as to the variability of the composition of butter-fat are obviously not based upon analyses of commercial butters made from the mixed milk of a number of cows. In connection with the adulteration of butter, "the more common offence" is, nowadays, not the sale of a factitious article, but rather the sale of mixtures of butter and margarine. The remark that "it is a common practice to mix a certain proportion of starch with yeast as a preservative" appears uncalled for, unless the book were written for defence advocates only. As to milk, the book takes us back to those bygone times in which the notorious lecture was delivered at the Health Exhibition, the Manchester milk case occurred, and the old Wanklyn process was in use, with limits fixed at 9 per cent. and 2·5 per cent. respectively.

The chemical part of the little work affords another proof of the good sense contained in the old adage, where the cobbler is advised to strictly confine his attention to his last. O. H.